

## A STUDY ON CHARACTERIZATION OF RADIATION INDUCED GRAPHENE OXIDE

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### ABSTRACT

*Interaction of graphene oxide and its related micro carbons through radiation exhibits various novel properties and phenomena. Irradiation of graphene oxide in solid state or in solution through Co-60, Pm-147, Ti-204 and <sup>90</sup>Sr-<sup>90</sup>Y gamma sources reduces it to graphene with negligible oxygen functionalities on the surface. This variation can be exploited for nano patterning and for large scale manufacturing of graphene oxide(GO). Gamma-induced dehydrogenation of hydrirogenated graphene can also be used for this radiation sensor purpose. All such gamma radiation induced transformations are associated with thermal effects. Results in a dramatic radiation sensor application. GO as well as graphene nanoribbons are excellent detectors of charged and uncharged particle radiation whereas has proven to be a GO is a good radiation detector.*

**KEYWORDS:** Hydrirogenated Graphene, Dramatic Radiation, Good Radiation

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### INTRODUCTION

Generally, graphene constitutes a single atom layer of thick carbon sheet. Graphene shows abundant interesting electronic, optical and mechanical properties due to its two-dimensional (2D) crystal shapes. The charge carriers of graphene flow ballistic in the 2D crystal lattice of graphene; subsequently it possesses high conductivity regardless of it being an natural compound [1]. Graphene is considered to be a building block because it can be wrapped up to form fullerene, rolled-up to form carbon nanotube or stacked up to form graphite. This arbitrary variation occurs in graphene exhibit phenonunal electrical properties. Graphene can be produced by the use of mechanical method as demonstrated by means of novoselov and andre geim. In which they concluded that this approach yields possess highest quality of graphene but minimal quantity compared to other methods<sup>2</sup>. An alternative method is required to mass produce graphene in mass amount since the mechanical is approach method yields low quantity which is suitable only for research purpose. One such alternative method named “chemical oxidation”.

Produces mass amount of graphene by using first introducing purposeful organizations which includes carbonyl, hydroxyl and peroxy in between carbon layers of graphite. These purposeful groups weaken the Van der Waals bond between the carbon layers which cause the graphene layers to peel off layer with the aid of layer from graphite<sup>3</sup>The ensuing graphene oxide (GO) may be reduced to graphene via adding hydrazine into the GO solution<sup>3</sup>. The criteria of batteries for electronics gadgets in latest days are lengthy-lastingness despite significant usage and rapid charging price during recharge (or) even as recharging. Therefore, super capacitors are

incorporated inside the batteries to abide with the criteria. These Electrochemical super capacitors are passive and have static electrical energy storage devices where extremely fast charging rate is considered to be a significant property the main property.

The properties of super capacitors comprises tremendous properties that includes high power capabilities, fast charge propagation, charge-discharge process occurring within seconds, long cyclic life, low maintenance, and low self-discharging<sup>4</sup>. Super capacitors also exhibit larger energy density compared to conventional capacitors. The electrodes present in the super capacitor are normally made of porous carbon but recently, the researchers start to investigate the possibilities of utilizing graphene as electrodes<sup>4</sup>. This is due to the cause that the graphene-based materials furnish shown interesting properties such as high surface area, high conductivity and capacitance and low production cost<sup>4</sup>. Universally a more prominent large area graphene is highly desirable as it significantly reduces high inter sheets contact resistance as compared with modest small area graphene<sup>5</sup>.

In this work, graphene oxide (GO) was synthesized using the simplified Hummer's method, which would yield GO with large lateral dimension in scalable quantity<sup>6</sup>. It was characterized using Ultraviolet (UV) and Fourier transform infrared spectroscopy. GO was used as an electrode material since it is the crucial derivative of graphene<sup>7</sup>. The capacitance measurements were performed at room temperature using different gamma ray emitting sources such that Co-60, Pm-147, Ti-204 and <sup>90</sup>Sr-<sup>90</sup>Yby connecting the sensing terminal to FTO and the signal terminal on the surface of GO layer. The sweeping rate for the gate voltage was varied from 1V to 5V/s for the C-V and IV measurements at different timings. Further, Go was utilized for the purpose of radiation sensor application.

## MATERIALS AND METHODS

### • Synthesis of GO

Go was synthesized from graphite powder consistent with the changed Hummer's technique. As an preliminary step, 2 g graphite powder become steadily brought into a hot 60ml concentrated H<sub>2</sub>SO<sub>4</sub> solution and stirred for 30 minutes. Further, 6 g of KMnO<sub>4</sub> was added pinch by using pinch underneath stirring which will avoid overheating and explosion after which it turned into left for every other 150 mins under stirring. Subsequently, 90 ml of distilled water was added drop by drop into the above mixture. After 15 minutes, 150 ml of distilled water and 5% of H<sub>2</sub>O<sub>2</sub> were added in order to terminate the reaction.

The product became washed with HCL (1:10) and then with water, and then suspended in distilled water. The brown dispersion become significantly dialyzed to put off residual metal ions and acids. After the unexploited graphite within the ensuing combination became removed with the aid of centrifugation, as-synthesized GO became dispersed into individual sheets in distilled water at a concentration of 0.47 mg/ml with the help of sonication and further it was characterized with UV, FTIR, XRD and Scanning Electron Microscopy(SEM)<sup>14-18</sup>.

### • Preparation of Electrodes

It is essential to observe that the working electrode is a critical variable in an electrochemical experiment. The running electrode is where the reactions of interest take vicinity and is normally constructed of an inert material. The ability of the working electrode is measured versus the reference electrode. A standard reference electrode is a acquainted saturated calomel electrode usually utilized in pH measurement. With the aid of layout, the reference electrode is continually at a acknowledged ability. Since potential is a measurement of the voltage difference between 2 points, the

fixed value of the reference electrode offers a manner to measure the working electrode capacity. In an electrochemical measurement system, current flows in the solution between the working and the counter electrodes. The counter electrode is usually a wire constructed of copper or other inert material.

- **Characterization**

The FT-IR spectra have been received on a Fourier Transform Infrared Spectrometer (EQUINOX 55, Bruker, Germany). The samples for FT-IR measurement have been prepared by means of grinding the dried powder of reduced graphene oxide. The grain size and surface morphology were observed by the field emission scanning electron microscope (FESEM). FESEM images of the Graphene Oxide (GO) have well defined and interlinked three-dimensional Graphene sheets, forming a porous network that resembles a free sponge like structure. X-ray diffraction, XRD, data were collected using a Rigaku D2000 Bragg-B0.rentano diffractometer, prepared with a copper rotating anode, diffracted beam monochromator tuned to radiation, and a scintillation detector. The entire data were collected using reflection mode  $\alpha$ Cu K geometry.

An LCR meter measures inductance (L), capacitance (C), and resistance (R). Relevant functions to search for in a LCR meter, capacitance meter are accuracy, test frequency, measured parameters, check voltage and test current. Statistics series and automation is made easy with a wide variety of interfaces which includes USB, host port, RS 232 and GPIB interfaces, that are to be had in almost all IET's LCR meters. Component and sensor testing frequently required for a lot more than simply a resistance, capacitance or inductance cost at given test frequency and stimulus voltage. An LCR meter must have the ability to measure more than one impedance parameters over a wide variety of frequencies and voltage.

- **LCR Meter Analysis on Gamma Radiation Induced GO**

The capacitance measurements were taken at room temperature using different gamma ray emitting sources such that Co-60, Pm-147, Ti-204 and  $^{90}\text{Sr}$ - $^{90}\text{Y}$  by connecting the sensing terminal to FTO and the signal terminal on the surface of GO layer. The sweeping rate for the gate voltage was varied from 1V to 5V/s for the C-V measurements at different timing 5- 30mins. The Go was used to radiation sensor application purpose.

## RESULTS AND DISCUSSIONS

The synthesized GO by Hummer's and Modified Hummer's methods are characterized by UV, X-Ray Diffraction Analysis (XRD), Fourier Transform- Infrared Spectroscopy (FT-IR), Field Emission Scanning Electron Microscopy FESEM and LCR measurement to capacitance.<sup>7-10</sup>.

- **UV Characterization**

Figure 3 depicts the ultraviolet-visible spectra of reduced graphene oxide and graphene oxide. The spectrum of graphene oxide has an absorption peak at 260 nm which is shifted to 300 nm in graphene. That is a red shift that is because of the electronic configuration in graphene within the reduction of graphene oxide. The absorption peak at 260 nm is attributed to transition of aromatic C-C ring. The UV spectra of reduced grapheme oxide on the other hand show the red shift at 260 nm. This absorption height is attributed to n- $\pi$  transition of C-O bonds now embedded with the aid of exfoliation and intercalation on the graphene.

- **FTIR characterization**

An FTIR spectrum evaluation includes in graph was accomplished to investigate the structure and functional groups of the substances, of graphene oxide and its reduced shape are depicted. Figure 4. The GO showed apparent adsorption bands for the carboxyl C=O ( $1755.31\text{ cm}^{-1}$ ), aromatic C=C ( $1385.09\text{ cm}^{-1}$ ), epoxy C–O ( $1086\text{ cm}^{-1}$ ), alkoxy C–O ( $1045\text{ cm}^{-1}$ ), and hydroxy –OH ( $3300.03\text{ cm}^{-1}$ ) groups. The presentation of oxygen-containing purposeful groups, consisting of C=O and C–O, similarly confirmed that the graphite certainly become oxidized into GO and became consistent with the literatures<sup>10–13</sup>. The presentation of C=C corporations showed that even graphite were oxidized into GO; the primary structure of layer graphite changed into nonetheless retained. The end result of FT-IR synthesis both similarly established the successful synthesis of GO.

- **Fesem (Field Emission Scanning Electron Microscope)**

Figure 5 depicts the FE-SEM images of the obtained GO flakes, with a film thickness of about  $4\mu\text{m}$ . The GO material that constitutes randomly aggregated, thin, crumpled sheets intently associated with each different to form a disordered solid.<sup>22</sup>

- **XRD**

In the XRD pattern of GO fig 7, peaks sturdy and sharp peak at  $2\theta = 11.7^\circ$  corresponds to an interlayer distance of  $7.6\text{ \AA}$  (d002). Reduced graphene oxide indicates a vast height that may be geared up by using a lorentz an feature into 3 peaks focused at  $2\theta = 20.17^\circ$ ,  $23.78^\circ$  and  $25.88^\circ$ , similar to interlayer distances of  $4.47$ ,  $3.82$  and  $3.53\text{ \AA}$ , respectively. This XRD result are related to the exfoliation and reduction tactics of GO and the procedures of disposing of intercalated water molecules and the oxide groups.

- **Gamma Radiation Induced Graphene Oxide**

The capacitance measurements were figure 8 depicts the capacitance level measurements of in radiated graphene oxide for different times intervals taken at room temperature preformed different gamma ray emitting sources Co-60, Pm-147, Ti-204 and  $^{90}\text{Sr}$ - $^{90}\text{Y}$ . By varying the sweeping rate for the gate voltage from  $1\text{V}$  to  $5\text{V/s}$  at different time intervals of 10mins, 20mins and 30mins. It is observed from the figure that capacitance levels get increased with the increase in time intervals, and also with the higher activity of gamma radiation. Thus, it has been found out that the higher activity source of CO-60 constitutes  $12,800\text{ dps}$  when compared to other lower activity sources of Pm-147, Ti-204,  $^{90}\text{Sr}$ - $^{90}\text{Y}$ , etc. Hence we analysed that CO-60 emission produces prominently a higher yield of capacitance which concludes that higher the activity of charged particle will results in higher production of capacitance.

- **IV Characterization**

The above i-v characteristic curves define the resistive element, in the sense that if we apply any voltage value to the resistive element, the resulting current is directly obtainable from the I-V characteristics. As a result, the power dissipated (or generated) by the resistive element can also be determined from the I-V curve.

If the voltage and current are positive in nature, then the I-V characteristic curves will be positive in quadrant I, if the voltage and therefore the current are negative in nature then the curve will be displayed in quadrant III as shown. In a pure resistance the relationship between voltage and current is linear and constant at a constant temperature, such that the current ( $i$ ) is proportional to the potential difference  $V$  times the constant of proportionality  $1/R$  giving  $i = (1/R) \times V$ . Then the current through the resistor is a function of the applied voltage and we can demonstrate this visually using an I-V

characteristics curve.

In this easy example, the cutting-edge  $i$  towards the capacity distinction  $v$ , is a straight line with regular slope  $1/r$  as the relation is linear and ohmic. But, realistic resistors may additionally exhibit non-linear behaviour under positive conditions for. example when exposed to excessive temperatures. There are many electronic components and devices that have non-linear traits, this is their  $v/i$  ratio is not consistent. Semiconductor diodes are characterized by using non-linear current voltage traits as the current flowing through a forward-biased commonplace silicon diode is restrained through the ohmic resistance of the PN-junction.

- **Ion Recombination**

Ion recombination means that the density of ions created in the sensitive media in an ionization chamber is diminished through charge reduction processes. These processes may lead back to the initial state of neutral molecules, thereby degrading the measurement signal. In the field of radiation dosimetry, ion recombination is commonly viewed as two separate processes, i.e. initial and general recombination. Initial recombination is considered as the interactions taking place between charges produced in the same incident ionizing particle track. General recombination is the process of neutralization of charge produced in different ionizing particle tracks. This process occurs during the charge migration to the respective electrodes under the influence of the collecting electric field given by the applied voltage to a graphene oxide based ionization chamber. When during radiation exposure time the graphene oxide will reduced and then voltage curve also shifted depending upon the gamma source activity and time.

In the recombination region (Region I), as voltage increases to  $V_1$ , the pulse peak will increase till it reaches a saturation value. At  $V_1$ , the sector strength among the cathode and anode is sufficient for collection of all ions produced within the detector. At voltages much less than  $V_1$ , ions pass slowly in the direction of the electrodes, and the ions tend to recombine to form neutral atoms or molecules. in this case, the pulse height is less than it might have been if all of the ions originally formed reached the electrodes.

## CONCLUSIONS

Modified Hummers method has been synthesize applied to prominent high area graphene oxide. This method was finished with the highest conversion stage of graphite flakes to graphene oxide and shows that pure graphene oxide is formed. XRD patter confirms the presence of graphene oxide to hexagonal structure.

The particle sizes were in the range of  $4\mu\text{m}$  to  $10\mu\text{m}$  from FESEM. FTIR confirms the formation of graphene oxide. The gamma radiation sensor is proved in this method, by demonstrating that the capacitance level increases with the increases in time exposure and high source activity. From this results, considered to be a GO is very effective gamma radiation sensor. The preliminary results make us believe that this new generation of radiation detectors will avail a wide number of applications. The preliminary results strongly support their feasibility as planar positive ion detectors in mass spectrometry, gas chromatography and ion mobility spectrometer<sup>19-20</sup>.

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## APPENDICES

### Figures

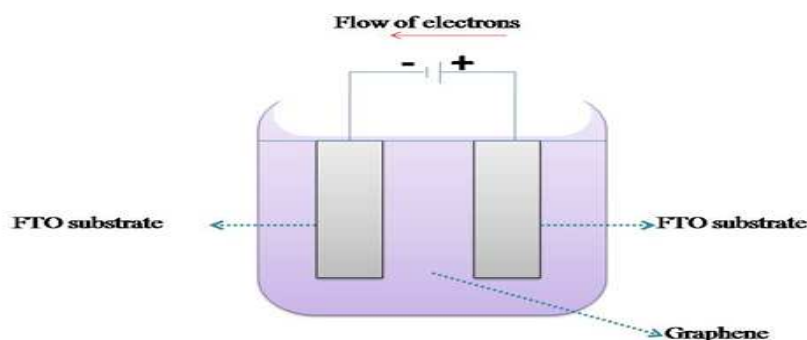


Figure 1: Schematic Diagram of Electrochemical Method for Grapheme Oxide

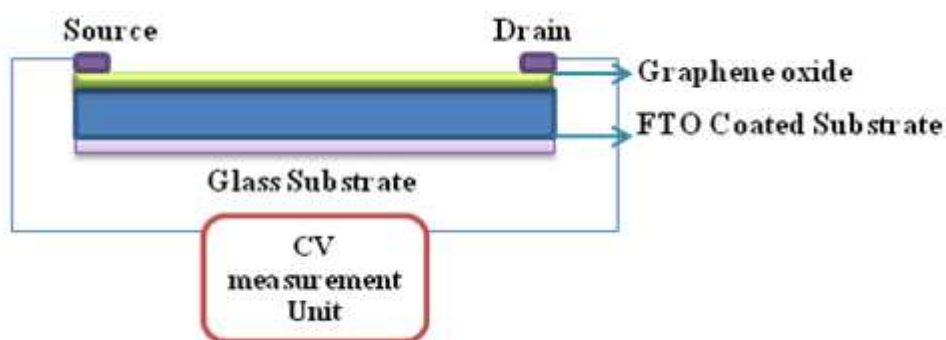


Figure 2: Schematic Diagram of That LCR Experimental Method

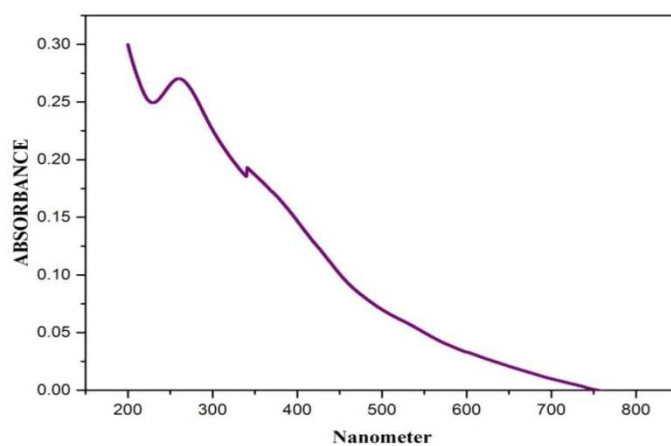


Figure 3: UV Vis Spectra of Graphene Oxide

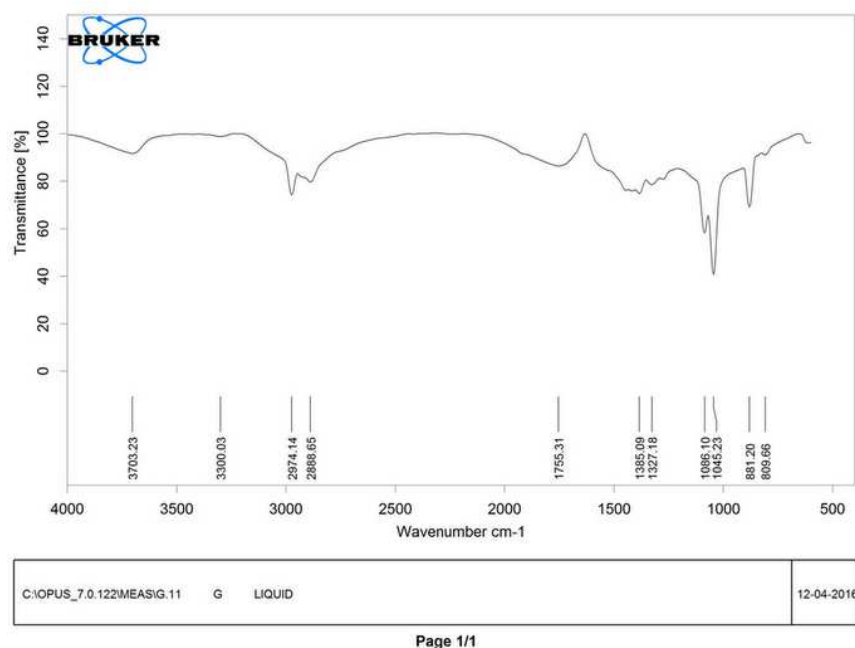
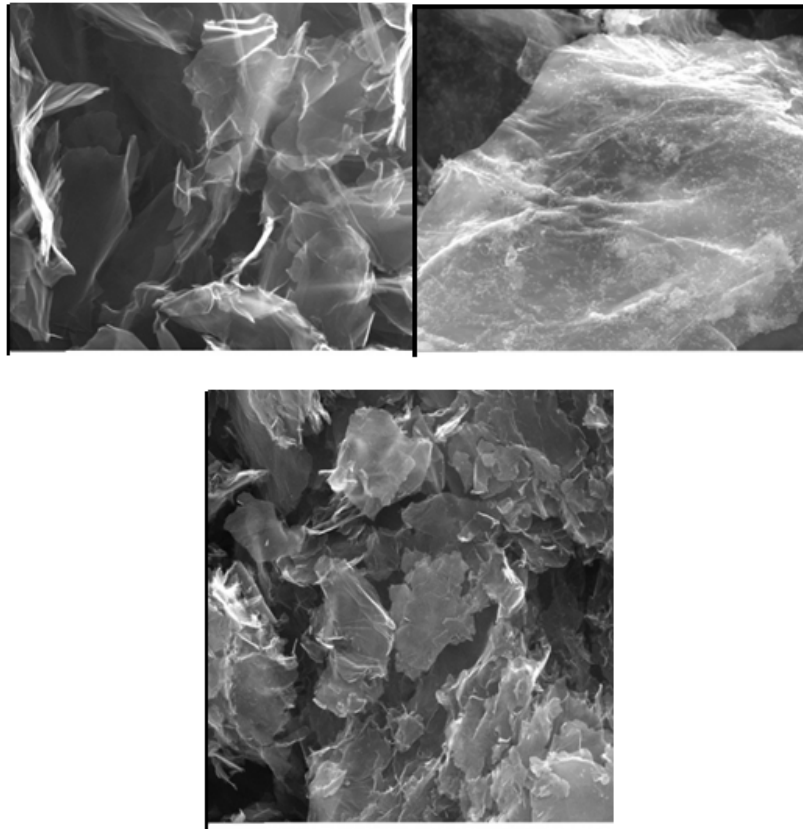
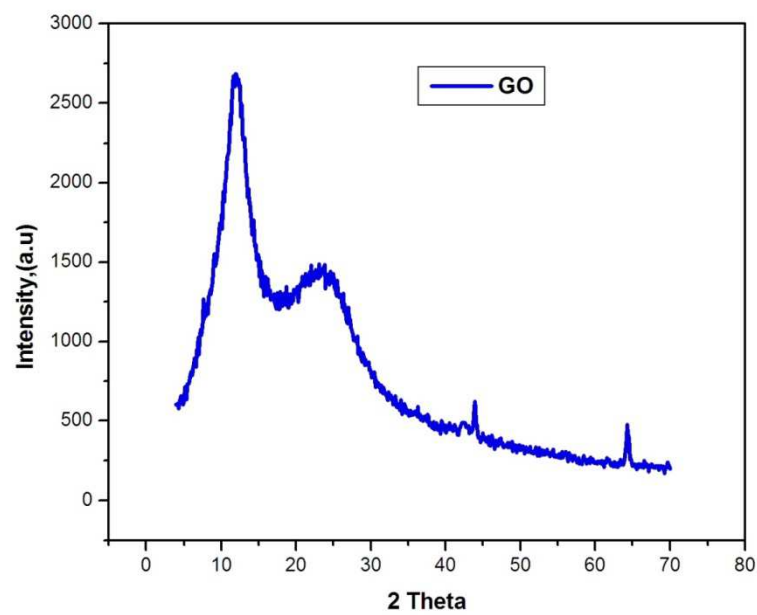


Figure 4: FTIR Spectra of Grapheme Oxide



**Figure 5: FE-SEM Image of Aggregated Graphene Oxide Flakes (A). FE-SEM Image of Graphene Oxide Flakes With Film Thickness of About 4 $\mu$ m (B)**



**Figure 6: XRD Spectra For GO**



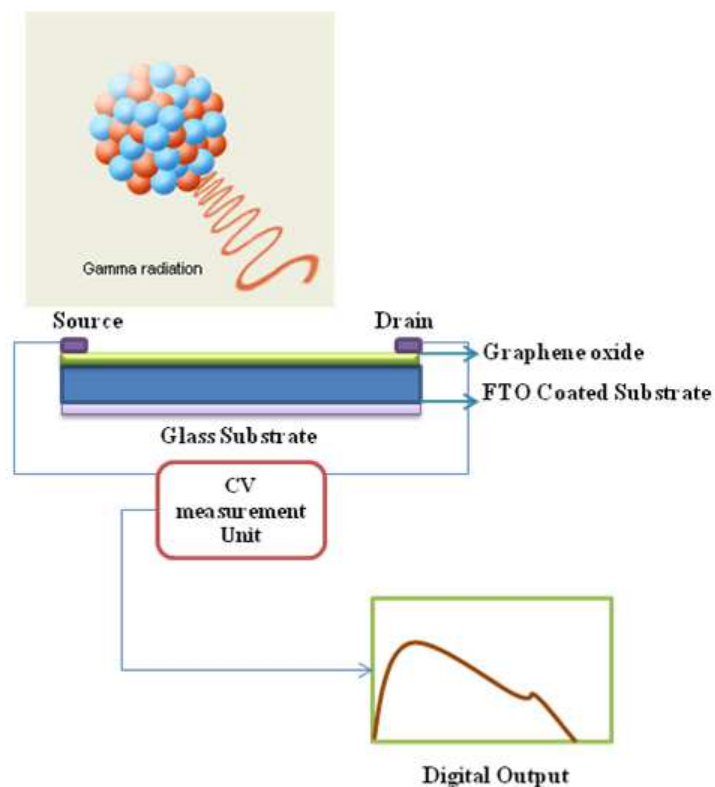


Figure 7: Shows That Experimental Setup for Gamma Radiation Induced Graphene Oxide

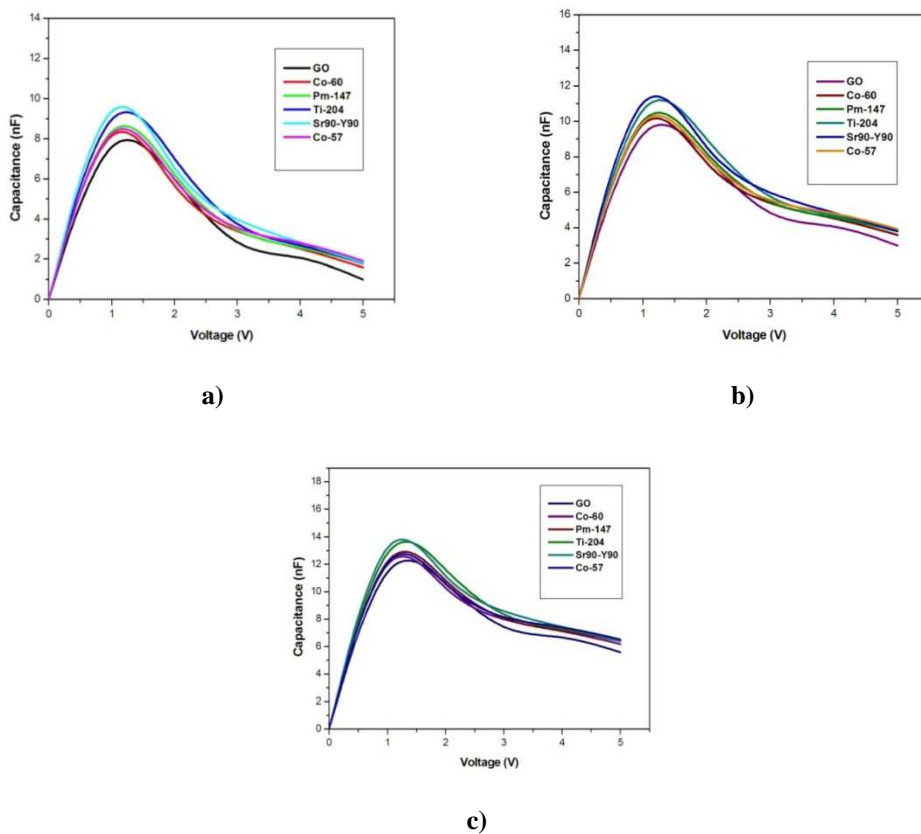
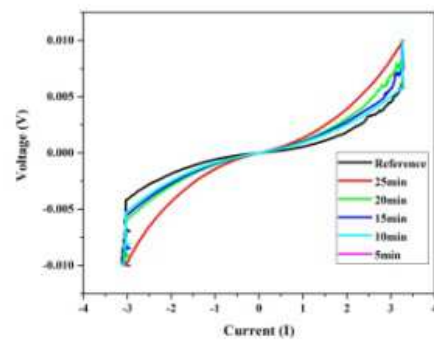
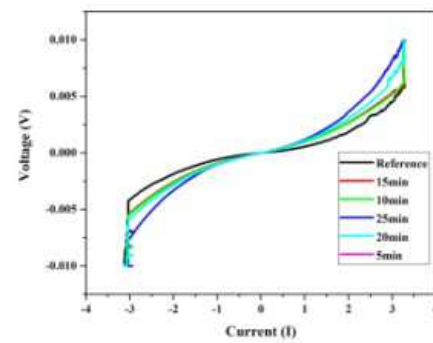


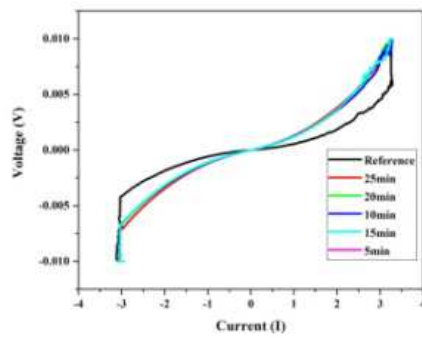
Figure 8: Graphical Representation of Gamma Radiation Induced Graphene Oxide Under The Exposure of A) 10mins B) 20mins C) 30mins



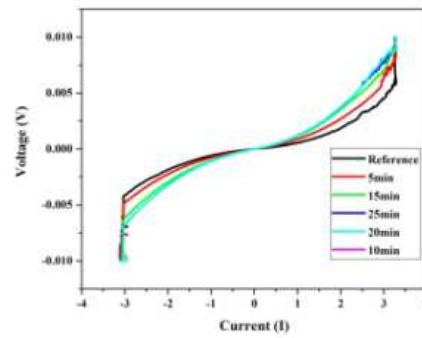
a) Co-60



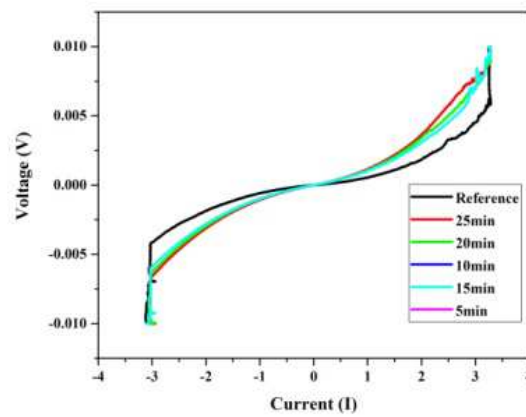
b) Sr90-Y90



c) Pm-147



d) Ti-204



e) Co-57

**Figure 9: Graphical Representation of Different Gamma Radiation Induced Graphene Oxide under the Exposure of 5 -25mins**